FEASIBILITY DEMONSTRATION OF PYROLYTIC

GRAPHITE COATED NOZZLES

RPL-TDR-64-176

Contract No. AF 04(611)-9708
United States Air Force (AFSC)
Rocket Propulsion Laboratory
Edwards, California

Third Quarterly Progress Report
Period Covered: 1 August through 31 October 1964

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FEASIBILITY DEMONSTRATION OF PYROLYTIC GRAPHITE COATED NOZZLES

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Project No. 3059 Program Structure No. 750G

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Period Covered: 1 August through 31 October 1964

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Prepared by: Atlantic Research Corporation
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ABSTRACT

The objective of this program is to demonstrate the feasibility of pyrolytic graphite coatings for use in uncooled solid propellant rocket nozzles (in a size range of use for practical propulsion units) under very severe operating conditions. In prior work at Atlantic Research Corporation, the excellent serviceability of pyrolytic graphite coatings in 1/2-inch diameter nozzles was demonstrated with propellants having flame temperatures from 5500°F to 6500°F. In the current work, nozzles of 1.1-inch and 2.3-inch diameter are to be tested with a 6550°F propellant. This report describes the work of the third quarter of this program.

Improvements were made in preparing crack-free sub-scale coated inserts. Flaw-free coatings as thick as 54 mils were prepared. Application of this improved art to full-scale inserts is underway. Stress analysis indicated the merit of alternate substrate materials and deposition work with these is underway.

A successful 35-second sub-scale firing was made but during a 60-second firing discrete coating losses were observed. In the first full-scale motor firing excessive erosion was observed from a coating containing a known separation. This behavior was analogous to early sub-scale experience.

1.0 INTRODUCTION

Pyrolytic graphite is a unique form of graphite which exhibits excellent erosion resistance in solid propellant rocket nozzles. The high density and absence of a binder phase are believed to explain its exceptional serviceability. Sub-scale rocket motor tests of 1/2-inch diameter pyrolytic graphite coated nozzles carried out at Atlantic Research Corporation have shown that consistently good performance can be achieved in nozzle service with propellant having flame temperatures from 5600°F to 6550°F. The higher the flame temperature and the motor operating pressure the higher the erosion rate observed for pyrolytic graphite. Specifically, 1/2-inch diameter nozzles with 6550°F propellant have shown average erosion rates of less than 0.5 mil/sec at 700 psi. The feasibility of scaling up to 1-inch and 2.3-inch throats with 6550°F propellant is to be determined under this program.

The best erosion resistance can be achieved by using pyrolytic graphite as a coating so that the layer plane surfaces are exposed to the combustion gas environment. To achieve the higher erosion resistance of the coating, the difficulties in maintaining coating integrity must be accepted and suitable designs must be demonstrated by test firings nozzles of useful size. To demonstrate the capabilities of pyrolytic graphite coated nozzles for solid propellant rocket motors operating under severe conditions, the current program consists of the design, fabrication and motor testing of a series of nozzles with an advanced propellant of 6550°F flame temperature. This report covers the third quarter of the program.

2.0 SUMMARY

During the third quarter, work was continued in three areas, stress analysis of pyrolytic graphite coated substrates, preparation of both sub-scale and full-scale nozzle inserts, and motor firing tests.

Stress calculations were extended to include the effect of nozzle curvature in the axial direction, but no principal stresses sufficient to explain the observed delamination cracks were found. The shear stresses associated with the presence of edges appear to be the most likely cause of the flaws noted. The effect of the substrate properties on the principal stresses (which lead to shear near the edges) was calculated. Reductions of stress through the use of low modulus, fibrous graphite or anisotropic high density graphite were indicated.

Through deposition process control crack-free sub-scale coated inserts more than 50 mils thick were prepared on both molded and low modulus, fibrous graphite substrates. Cracks were observed in full-scale coated inserts at coating thicknesses comparable to those in the sub-scale units. Bond line failures were more prevalent so that an improved pretreatment procedure was adopted. It is anticipated that the process improvements and more suitable substrates defined in sub-scale and analysis work can be used to improve the full-scale coatings.

Three motor firing tests were carried out. A sub-scale test (SS-3) of a thin crack-free coating showed an acceptable erosion rate of 0.55 mil/sec in a 35-second firing. In a full duration, 60-second, sub-scale firing (SS-4) of a thicker crack-free coating partial coating loss was noted at 29-second burning time. Accelerated erosion and additional discrete loss of coating led to complete removal of the coating during the 65-second total firing time. This partial loss of a crack-free coating after 29 seconds is the most serious failure to date indicating the need for additional control of the stress levels in the coating. A full scale test (FS-1) of an insert with a known separation at the exit end showed excessive erosion but without discernible spallation in a fashion very similar to test SS-1 in which the coating had a similar flaw.

3.0 PROGRAM RESULTS

3.1 Stress Analysis

To investigate the effect of axial nozzle curvature on the stresses in a coated insert section calculations were made for a composite cylinder with the coating on the outside. The actual nozzle shape is a section of a torus surface. Thus, previous calculations for a cylindrical composite shape with the coating on the inside represented an idealization of the effect of nozzle throat radius on coating stresses. Looking at a coating on the outside of a cylinder is equivalent to straightening out the toroidal section and treating the nozzle radius of curvature as the cylinder radius. A total of twelve cases were calculated including the effect of two radii of curvature two thicknesses of a molded graphite substrate, and three pyrolytic graphite coating thicknesses. The maximum tensile stresses in each principal direction in both the coating and the substrate are listed in Table I. No principal stresses sufficient in magnitude to be considered the conclusive source of the observed delamination flaws were found, but those data do help to indicate the effects of the geometrical factors.

In an effort to identify preferred substrate graphites to reduce the stresses from thermal expansion mismatch, a series of calculations were made for a molded graphite (ATJ), an anisotropic, high density graphite (ZTA), and a low modulus fibrous graphite (PT-0114). The thermal expansion data for each of these types of graphite were taken from the most recent and complete source available, namely Air Force Report WADD TR61-74, Volume XXVI, which reports property data on newly developed grades of graphite. The data for ATJ, which are included as a reference standard, are somewhat different from those taken from the Industrial Graphite Engineering Handbook (National Carbon Company). A detailed review of the data on pyrolytic graphites also led to a modification of the values used earlier. The stress levels in both substrate and coating layers were calculated for composite cylinders of two diameters and three coating thicknesses for each of the three substrate materials. The results are summarized in Table II. The effect of substrate properties is apparent from examination of this table. The significant

Table I. Stresses in Composite Cylinder with Coating on Outside

Conditions: Molded substrate properties and coating properties as shown in QPR Number 2.

Coating Thickness (mil)	Maximum Tensile Stresses in Coating			Maximum Tensile Stresses in Substrate		
	Radial (psi)	Hoop (psi)	Axial (psi)	Radial (psi)	Hoop (psi)	Axial (psi)
A. Substrate radius = 1.600"; substrate thickness = 0.100"						
30	277	a	a	277	4440	2710
50	384			384	6210	3860
80	500			500	7900	5080
B. Substrate radius = 1.600"; substrate thickness = 0.400"						
30	382			382	1900	1050
50	610			610	2710	1580
80	925			925	3840	2290
C. Substrate radius = 3.200"; substrate thickness = 0.100"						
30	134			134	4300	2650
50	182			182	5820	3760
80	232			232	7370	4890
D. Substrate radius = 3.200"; substrate thickness = 0.400"						
30	188			188	1680	910
50	294			294	2470	1400
80	431			431	3510	2070

[a] Where no value is listed, stresses are entirely compressive.

Table II. Effect of Substrate Properties on Stresses in Composite Cylinders

Conditions: PG expansion to 2000°C, with grain = 0.0049; across grain = 0.0500
ATJ expansion to 2000°C, with grain = 0.00772; across grain = 0.01029
ZTA expansion to 2000°C, with grain = 0.00587; across grain = 0.01827
PT-0114 expansion to 2000°C, with grain = 0.00528; across grain = 0.00714

Inside Diameter (inch)	Coating Thickness (mil)	Maximum Tensile Stresses in Coating: Radial (psi)	Hoop (psi)	Axial (psi)	Maximum Tensile Stresses in Substrate: Radial (psi)	Hoop (psi)	Axial (psi)
A. ATJ substrate, 0.700" thick							
1.120	30	1.	638	a	a	322	567
1.120	50	93.	6010			262	900
1.120	80	425.	13,200		18		1310
2.300	30					380	680
2.300	50					505	1,070
2.300	80	18	2,670			565	1,560
B. ZTA substrate (anisotropy same orientation as PG); 0.700" thick							
1.120	30			1,680		3,770	
1.120	50					5,360	23.
1.120	80					6,880	154.
2.300	30			2,220		3,040	
2.300	50			655.		4,580	
2.300	80					6,360	124.
C. PT-0114 substrate; 0.700" thick							
1.120	30	111.	6,830		74		189.
1.120	50	271	10,800		154		256
1.120	80	608	16,500		280		310
2.300	30	22	2,980		9		220
2.300	50	61	5,050		28		297
2.300	80	151	8,040		66		363

[a] Where no value is listed, stresses are entirely compressive.

reduction in stresses for a low modulus, fibrous graphite substrate is particularly interesting. The suitability of such substrate material will be explored in deposition work and motor testing.

3.2 Deposition Work

Preparation of coated insert sections of both sub-scale and full-scale size was carried out on a continuing basis throughout the quarter. The improvements achieved for each size are outlined separately in the following sections.

3.2.1 Sub-Scale Inserts

A steady advance in our capability to prepare flaw-free coatings was made in the sub-scale system. The first flaw-free coated section prepared and fully machined for firing had a coating thickness of approximately 23 mils at the throat. This insert was tested in the third sub-scale firing (SS-3) discussed below. Following the preparation of this relatively thin, crack-free coating experimental work continued and coatings free of cracks as thick as 54 mils were successfully deposited on molded graphite substrates. The principal process changes responsible for this improvement were geometrical changes in the substrate and deposition chamber and more adequate thermal pretreatment of the substrates. The upper limit of coating thickness which can be made without cracking has not yet been determined for the current deposition technique.

Following the analysis of the stress conditions for different substrate materials, an experimental effort was made to coat fibrous graphite (low modulus) substrates. Coatings of nominal 50 and 80 mil thickness were prepared without delamination cracking. No visible separations or cracking was noted on the substrate in spite of its low cross grain strength. Again, the upper limit for crack-free conditions was not determined since coatings of adequate thickness were prepared.

3.2.2 Full-Scale Inserts

Less work was carried out on full-scale inserts since those represent a scale-up effort intended to apply sub-scale experience to hardware suitable for larger motors. Delamination cracking was observed on full-scale inserts at coating thicknesses of the same order as those which led to cracks in the

sub-scale inserts. This was somewhat surprising because, in general, the principal stresses caused by anisotropy are a function of the ratio of coating thickness to radius of curvature rather than to the absolute thickness value. Thermal expansion mismatch factors are more nearly dependent on the linear measurements of the various portions of a composite so that the observed behavior appears to indicate a predominate role of expansion mismatch stresses. Another factor worth note was the qualitative difference in the nature of the cracking observed in full-scale inserts compared to the sub-scale units. On the full-scale units cracking appeared largely at or very near the substrate-coating interface. On the sub-scale inserts the cracks were generally completely within the coating, most often two-tenths or more of the coating thickness away from the interface.

The observations described above caused an emphasis to be placed on two factors in full-scale deposition work. First, more extensive thermal pretreatment of substrates was adopted to prevent any surface effects during heat-up which would be detrimental to the bond strength between the coating and the substrate. Second, a renewed effort was made to reduce the incompatibility of the substrate and coating by considering different substrate graphites. At the end of the current quarter, work on these improvements was underway. Results since that time have indicated considerable success. These data will be reported after the current work is completed.

3.3 Motor Firing Tests

Three nozzles were tested in motor firings during this quarter. These three tests, which included the third and fourth sub-scale firings (SS-3 and SS-4) and the first full-scale firing (FS-1), are discussed below.

3.3.1 Sub-Scale Firings

The third sub-scale test (SS-3) was the first firing in which the pyrolytic graphite coating showed no visible cracks on microscopic examination of the polished section at each end of the insert. At the time of this firing, the 23.5 mil coating thickness on this insert (No. 357-17) was the thickest crack-free coating deposited on a molded graphite substrate. The erosion rate, calculated from the measured throat diameters before and after

firing, was 0.55 mil/sec. Although this firing was of only 35 seconds duration, it demonstrated the basic serviceability of a pyrolytic graphite coating when coating integrity is maintained. Due to the marginal thickness of the coating it was essentially destroyed at the throat section but the inlet portion of the coating remained to control nozzle erosion. The data from this test is included in Table III and a reproduction of the motor pressure trace is shown in Figure 1.

After further deposition work had resulted in the preparation of thicker coatings without visible cracks, another sub-scale firing was made. This firing which was an attempt to achieve a successful full duration (60-second) test in the sub-scale system, utilized a 54 mil thick coating on a molded graphite substrate (No. 357-32). The performance of this insert was disappointing. Discrete losses of coating occurred first at 29.5 seconds and again at 37 seconds. A final loss appears to have taken place at approximately 60 seconds. Data from this test are in Table III and the motor pressure trace is Figure 2. From calculations based on ballistic characteristics of the propellant, the erosion rate of the coating prior to the first discrete loss of coating appears to have been about 0.7 to 0.8 mils/sec although this calculation is necessarily approximate. The over all erosion rate, based on before and after test measurements was 1.8 mils/sec. It must be concluded that in this firing the cumulative effect of residual stresses from deposition and thermal stresses generated during firing caused progressive coating failure. One immediate means of reducing these effects is to reduce the stress problem by a more favorable choice of substrate. This approach is discussed above in the report on the deposition studies.

3.3.2 Full-Scale Firings

The first full-scale firing was made during the quarter. A 42-mil thick coating on molded graphite (No. 357-27) which contained a visible microscopic separation of the coating from the substrate at the exit end was tested. Because the nature of the flaw in this insert was quite different from those found in sub-scale units, a test of the effect of such a flaw on full-scale nozzle performance was indicated.

Table III. Summary of Motor Firing Data

Program No.	Firing Designation	Duration (sec)	Motor Pressure Max. (psi)	Motor Pressure Ave. (psi)	Nozzle Diameter Before (inch)	Nozzle Diameter After (inch)
SS-3	EPb-3	34.8	714	618	1.136	1.173/1.176
SS-4	EPb-5	64.9	812	590	1.111	1.330/1.365
FS-1	EPb-4	36.6	723	623	2.316	2.429/2.493

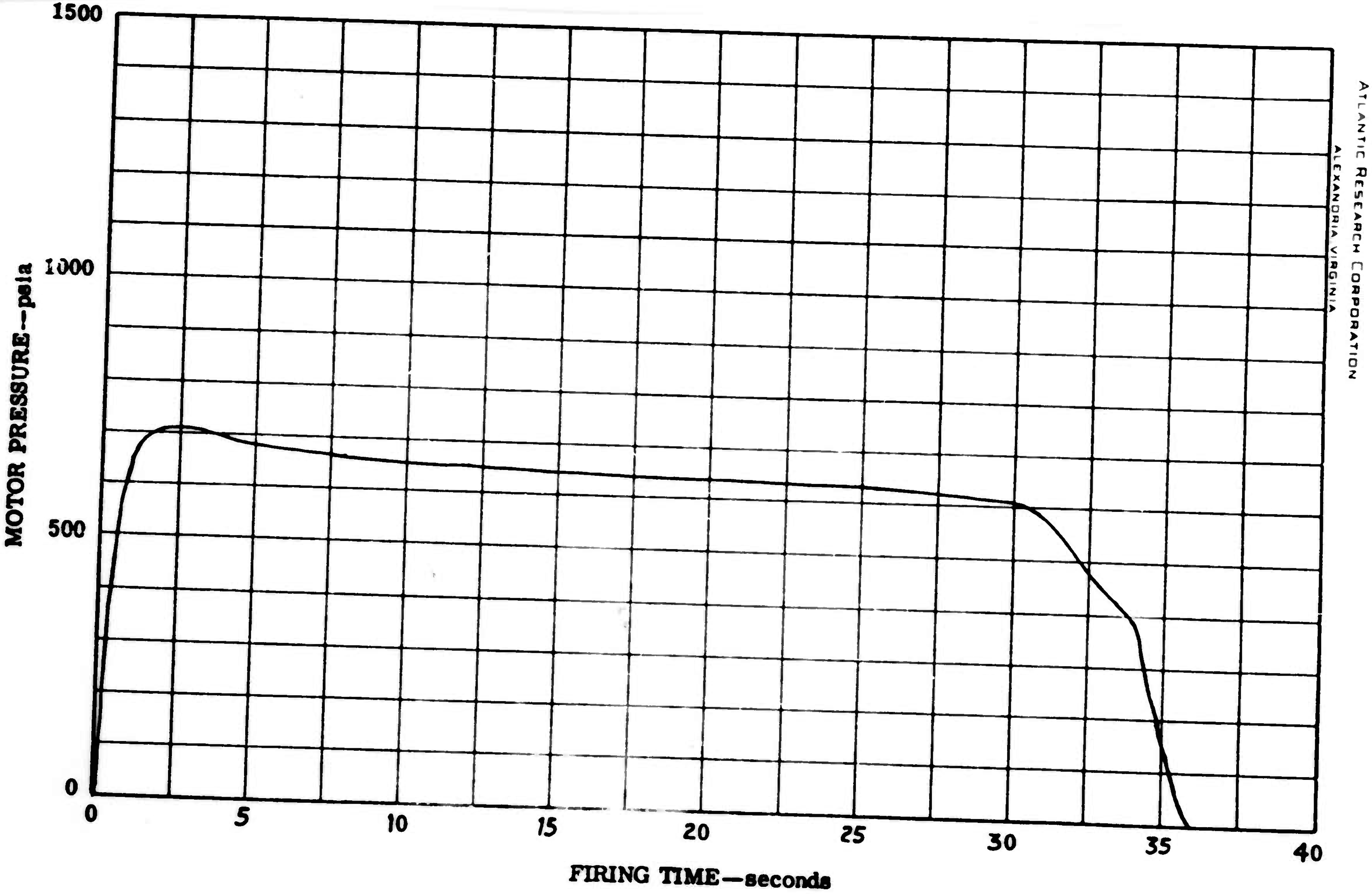

Figure 1 . Motor Pressure Trace for Firing EPb-3 (SS-3) Sub-Scale

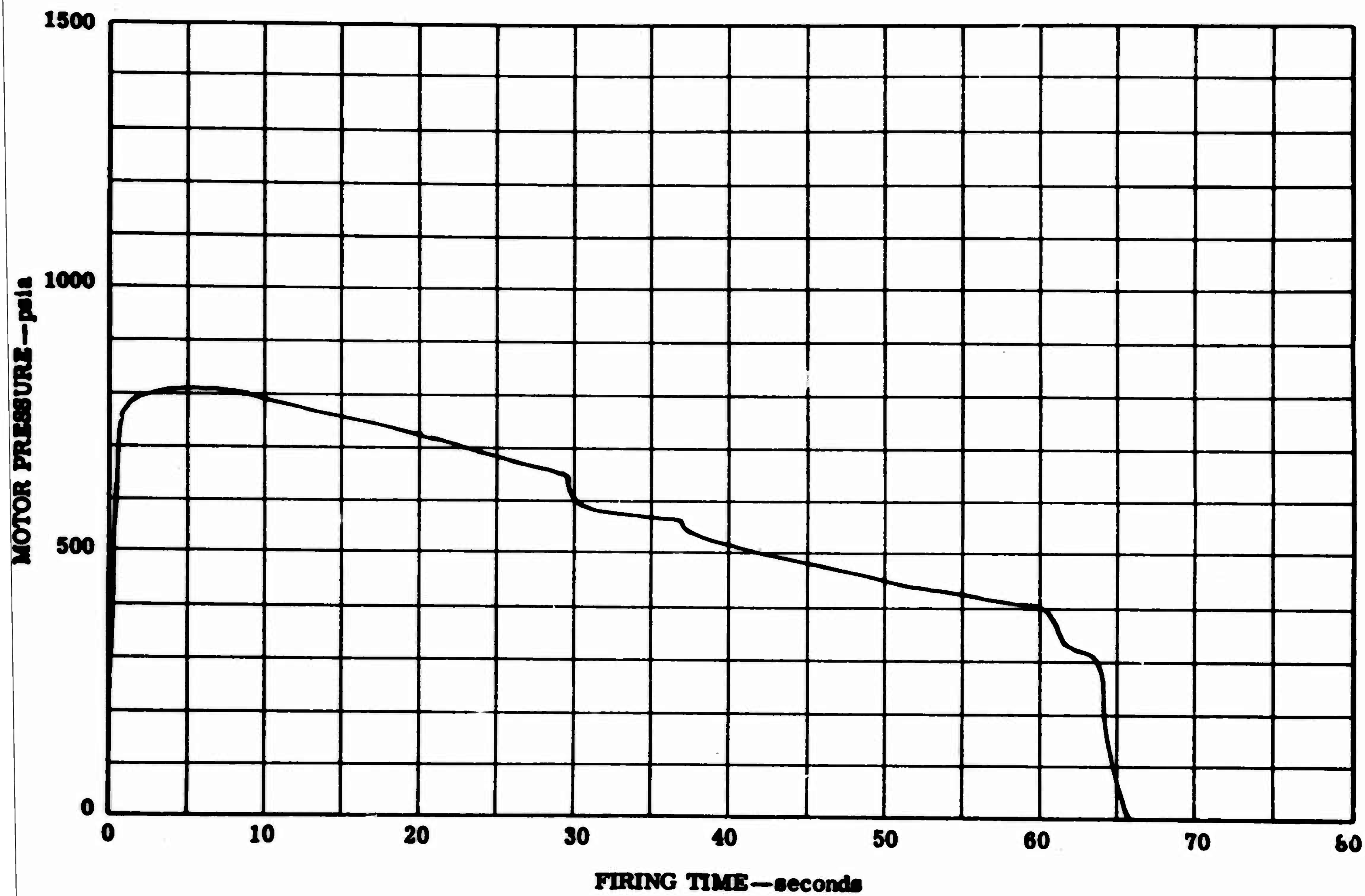

Figure 2 . Motor Pressure Trace for Firing EPb-5 (Sub-Scale 4)

The performance of this cracked full-scale insert was quite similar to results obtained earlier in sub-scale test of an insert with a crack at the exit end. The over all erosion rate, based on before and after diameter measurements, was somewhat over 2 mils/sec. A steady but excessive erosion pattern was indicated by the motor pressure trace, shown in Figure 3. The data from this test are included in Table III.

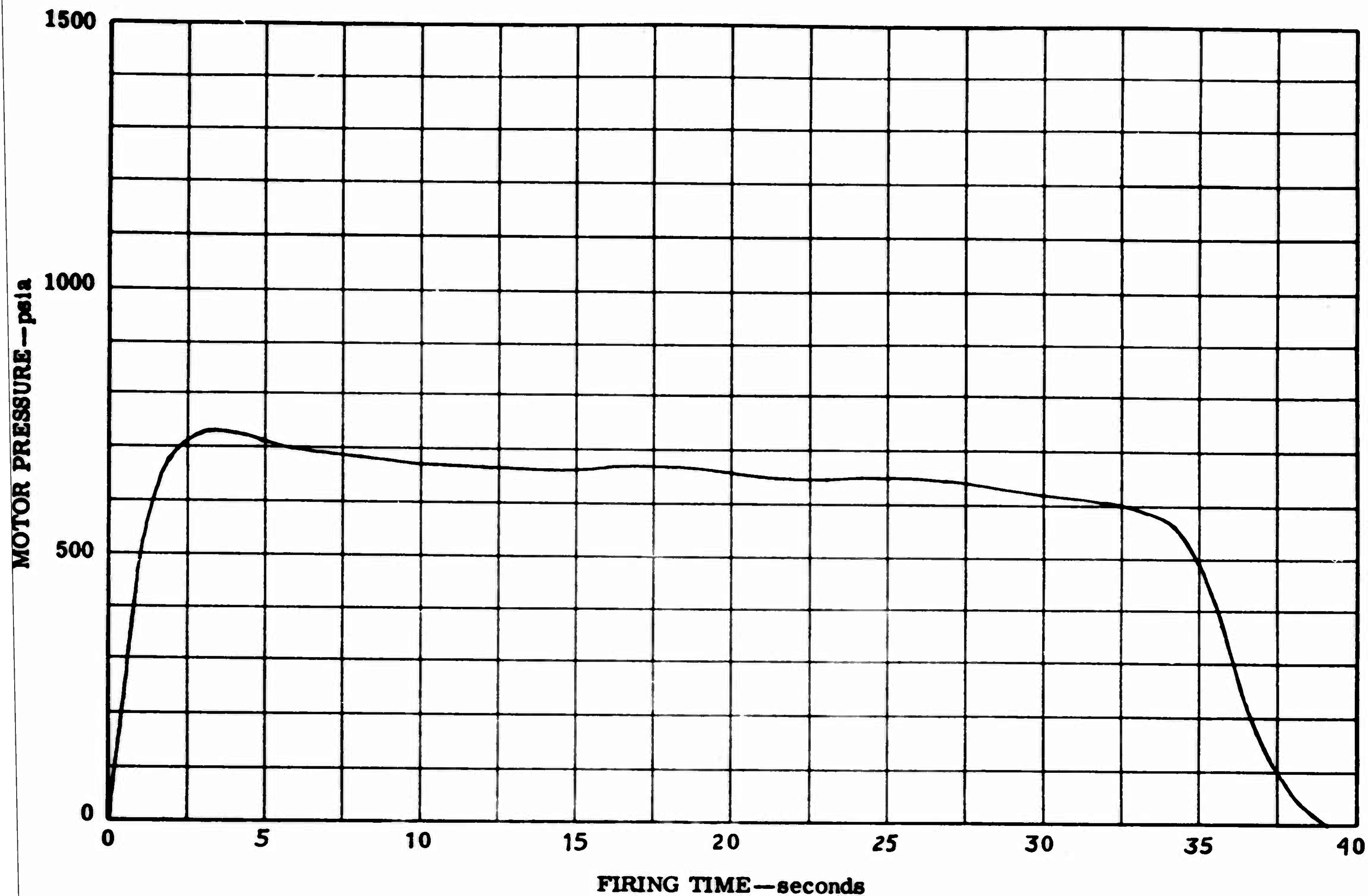

Figure 3 . Motor Pressure Trace for Firing EPb-4 (FS-1) Full Scale

4.0 FUTURE WORK

Deposition work is continuing in an effort to prepare thicker deposits free of cracks and serviceable under the severe motor test conditions selected for this program. Further motor tests should be scheduled based on reasonable technical expectations. The remaining sub-scale firing (SS-5) included in the current program schedule will be used to test a coating deposited on a low modulus fibrous graphite. It seems apparent that some reduction in the severity of the remaining full-scale firings will be necessary to maintain technical merit since the degree of success to date does not justify rapid advance to 100 second demonstration firings of full-scale nozzles. The proper schedule modifications are under current discussion with the Air Force.

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Improvements were made in preparing crack-free sub-scale coated inserts. Flaw-free coatings as thick as 54 mils were prepared. Application of this improved art to full-scale inserts is underway. Stress analysis - indicated the merit of alternate substrate materi- als and deposition work with these is underway.

A successful 35-sec sub-scale firing was made but during a 60-second firing discrete coating losses were observed. In the first full-scale motor firing erosion was observed from a coating con- taining a known separation. This behavior was analogous to early sub-scale experience.

1. Rocket Nozzle Material
2. Pyrolytic Graphite

I. AFRPL Project No. 3059

II. Contract No. AF 04(611)-9708

III. Atlantic Research Corporation Alexandria, Virginia

IV. Dr. J. D. Batchelor

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